

# PATENT ABSTRACTS OF JAPAN

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(71)Applicant : SUMITOMO BAKELITE CO LTD

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(72)Inventor : ASAUMA SUMITOSHI  
MORI TETSUYA

**(54) SEALING MATERIAL COMPOSITION FOR LIQUID CRYSTAL DISPLAY ELEMENT, PRODUCTION OF LIQUID CRYSTAL DISPLAY ELEMENT AND LIQUID CRYSTAL DISPLAY ELEMENT**

(57)Abstract:

**PURPOSE:** To improve productivity and yield by consisting essentially of a thermosetting resin, a radiation and/or light polymerizable monomer, a hardening agent and an inorganic filler and incorporating no solvent, which is not concerned in the hardening reaction.

**CONSTITUTION:** A sealing material composition for liquid crystal display element consists essentially of the thermosetting resin, the radiation and/or light polymerizable monomer, the hardening agent and the inorganic filler and does not incorporate any solvent which is not concerned in the hardening reaction. The radiation and/or light polymerizable monomer is allowed to react by irradiating with radiation and/or light after the sealing material composition is applied on one side of a substrate for liquid crystal display element and after that, the other substrate is laminated and the thermosetting resin is allowed to react and hardened by heating. As the radiation and/or light polymerizable monomer, acrylic acid, an acrylic ester, methacrylic acid, a methacrylic ester, acryl amide, a vinyl ester, styrene and a monomer containing  $\geq 1$  epoxy group or the like in the molecule are exemplified.

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**CLAIMS****[Claim(s)]**

**[Claim 1]** The sealant constituent for liquid crystal display components which uses thermosetting resin, a radiation and/or a photopolymerization nature monomer, a curing agent, and an inorganic filler as an indispensable component, and is characterized by not including the solvents which do not participate in a hardening reaction.

**[Claim 2]** The sealant constituent for liquid crystal display components according to claim 1 whose thermosetting resin is an epoxy resin.

**[Claim 3]** The sealant constituent for liquid crystal display components according to claim 1 a radiation and/or whose photopolymerization nature monomer are radical polymerization nature and/or cationic polymerization nature.

**[Claim 4]** The manufacture approach of the liquid crystal display component characterized by making a radiation and/or a photopolymerization nature monomer react, piling up another substrate after that and making thermosetting resin react by heat hardening by irradiating a radiation and/or light after the sealant constituent for liquid crystal display components given in any 1 term of claims 1-3 is applied to one side of the glass for liquid crystal display components, or a plastic plate.

**[Claim 5]** The liquid crystal display component using the sealant constituent for liquid crystal display components given in any 1 term of claims 1-3.

**[Claim 6]** The liquid crystal display component manufactured by the manufacture approach of a liquid crystal display component according to claim 4.

**DETAILED DESCRIPTION****[Detailed Description of the Invention]****[0001]**

**[Industrial Application]** This invention relates to the liquid crystal display component which used the manufacture approach of the sealant constituent for liquid crystal display components, and a liquid crystal display component, and it.

**[0002]**

**[Description of the Prior Art]** In recent years, the liquid crystal display component has spread widely from the descriptions, such as a light weight, a thin shape, and a low power. Although the liquid crystal display component has structure which put liquid crystal with the substrate of glass or plastics, it needs to close a perimeter with adhesives so that inner liquid crystal may not begin to leak outside, and, generally is calling the adhesives the sealant for liquid crystal display components (omitting liquid crystal sealant).

**[0003]** Although the thermosetting large epoxy resin is used for current and this liquid crystal sealant, the liquid crystal sealant which was excellent for the productivity of a liquid crystal display component, the improvement in the yield, or the improvement in dependability of a liquid crystal display component is called for.

**[0004]** For example, in the production process of a general liquid crystal display component, in order to apply a liquid crystal sealant to one side of a substrate by screen-stencil or the dispenser and to remove the solvent contained, the heating process of prebaking is performed at the temperature of 50 to about 100 degrees C, another substrate is piled up after that and this hardening is performed at the temperature of 100 to 200 degrees C. However, at the prebaking process for removing a solvent, the solvent which remains in the sealant by which a solvent is not removed completely and

actual hardening was carried out may be eluted into liquid crystal, and may become the cause of reducing a display property. Moreover, generally it is required for a prebaking process from 10 minutes for 60 minutes, and shortening this time amount is also called for.

[0005]

[Problem(s) to be Solved by the Invention] The liquid crystal display component using the manufacture approach of the sealant for liquid crystal display components and a liquid crystal display component and it which the productivity of a liquid crystal display component and the yield of this invention improve compared with the conventional liquid crystal sealant, and are excellent in the dependability of a liquid crystal display component is offered.

[0006]

[Means for Solving the Problem] This invention Thermosetting resin, a radiation and/or a photopolymerization nature monomer, a curing agent, The sealant constituent for liquid crystal display components which uses an inorganic filler as an indispensable component and is characterized by not including the solvents which do not participate in a hardening reaction, And after said sealant constituent was applied to one side of the substrate for liquid crystal display components, A radiation and/or a photopolymerization nature monomer are made to react by irradiating a radiation and/or light. It is about the manufacture approach of the liquid crystal display component characterized by piling up another substrate after that and making thermosetting resin react by heat hardening, and the liquid crystal display component manufactured by the above-mentioned manufacture approach using the above-mentioned sealant constituent.

[0007] Although especially the thermosetting resin used by this invention is not limited, when an epoxy resin is used, a good property is acquired especially. It is not limited especially as an epoxy resin. Generally The bisphenol A mold epoxy resin, An alkylation bisphenol A mold epoxy resin, a bisphenol female mold epoxy resin, An alkylation bisphenol female mold epoxy resin, a bisphenol smooth S form epoxy resin, An alkylation bisphenol smooth S form epoxy resin, a phenol novolak mold epoxy resin, A cresol novolak mold epoxy resin, a biphenyl mold epoxy resin, A naphthalene mold epoxy resin, a glycidyl amine mold epoxy resin, a dicyclopentadiene mold epoxy resin, a silicone modified epoxy resin, an urethane modified epoxy resin, a rubber modified epoxy resin, etc. are used. these -- a kind -- or two or more sorts are used, using together.

[0008] Moreover, although not limited especially as a radiation and/or a photopolymerization nature monomer, the polymerization nature monomer by the cation initiation reaction according [ a polymerization reaction ] to radical polymerization nature or a radiation, and/or light is suitable. the monomer which contains an acrylic acid, acrylic ester, a methacrylic acid, methacrylic ester, acrylamide, vinyl ester, styrene, one or more epoxy groups, etc. in intramolecular as those examples -- it is -- these -- a kind -- or two or more sorts are used, using together. Furthermore, a suitable initiator can be used together in the polymerization reaction by the radiation and/or light.

[0009] Moreover, although especially the curing agent of thermosetting resin is not limited, either, generally an amine system curing agent, an imidazole system curing agent, a dicyandiamide, the Hydrazide system curing agent, an acid-anhydride system curing agent, a phenol system curing agent, etc. are usable, in order to raise shelf life further, these microencapsulation curing agents, an adduct mold-curing agent, etc. are usable, and a hardening accelerator can also be further used together. Generally as a hardening accelerator, an amine system compound, the Lynn system compound, an imidazole system compound, a urea system compound, etc. are used.

[0010] Furthermore, it is the purpose which prevents reservation of printing workability, turbulence of a printing pattern, etc., and inorganic fillers, such as the carbonate of various metals, a sulfate, a silica, an alumina, titanium oxide, and potassium titanate, are used for the sealant constituent for liquid crystal display components of this invention, and the range of 2 to 60 weight section is suitable for it to the pitch 100 weight section as the addition.

[0011] Moreover, the solvents which do not participate in a hardening reaction are not contained in the sealant constituent for liquid crystal display components of this invention. Therefore, the poor display of the liquid crystal display component by the solvent which often poses a problem by the conventional liquid crystal sealing compound and which remains in a hardened material oozing out in liquid crystal is not caused.

[0012] Furthermore in this invention, a coupling agent, a defoaming agent, a leveling agent, etc. may be added if needed other than the aforementioned thermosetting resin which is an indispensable component, a radiation and/or a photopolymerization nature monomer, a curing agent, and an inorganic bulking agent.

[0013] In using the sealant constituent for liquid crystal display components of this invention, and manufacturing a liquid crystal display component, the process shown below is taken. First, the polymerization of a radiation and/or the photopolymerization nature monomer is carried out by forming a seal pattern in either the glass for liquid crystal display components, or the substrate of plastics by screen printing, the dispenser applying method, etc., and irradiating a radiation and/or light as usual. Although most fluidities of a sealant are lost in this condition, the thermosetting resin contained in a sealant remains in the unreacted condition. Next, thermosetting resin is made for another substrate to react lamination and by heating, and two substrates are pasted up.

[0014] The description of this manufacture approach makes tuck nature discover [ sealant / containing the conventional solvent / liquid crystal ] in a room temperature or an about 60-degree C temperature region by vaporizing a solvent to some extent by prebaking after seal pattern formation. By this invention, grant of the tuck nature by the solvent vaporization by the conventional prebaking is performed by carrying out the polymerization of a radiation and/or the photopolymerization nature monomer to fixing two substrates, where another substrate is stuck. As the advantage, since a solvent is not included, from several seconds, I hear that the polymerization of that there is no problem of a residual solvent, a radiation, the radiation by light, and/or a photopolymerization nature monomer can carry out in a short time of several minutes, and it occurs. Moreover, although reinforcing with UV hardening resin as the solution depending on a sealant constituent or prebaking conditions although tuck nature may be inadequate in case the conventional liquid crystal sealant is used is performed, in this invention, such a process is also unnecessary.

[0015] Furthermore, since the boiling point is generally a high boiler 100 degrees C or more, in order to vaporize a solvent as much as possible, as for the solvent currently used for the liquid crystal sealant containing the conventional solvent, it is desirable but to prebake at an elevated temperature as much as possible, and on the other hand, if it prebakes at an elevated temperature, the problem that hardening of resin will also advance will arise. More, hardening of the sealant in low temperature follows this problem on asking, and it becomes what has much more difficult solution. when using plastics for a substrate especially, compared with glass, it is markedly alike, and since it is low, the thermal resistance of a substrate poses a big problem. At this point, this invention without the need for prebaking also has the description that it is easy to make curing temperature of thermosetting resin into low temperature in the range which does not have a problem in the shelf life of a sealant constituent.

[0016]

[Example] Although the example of this invention is explained below, this invention is not limited at all by these examples.

[0017] (Example 1) It is as thermosetting resin as the bisphenol A mold epoxy resin (oil-ized shell epoxy company make, Epicoat 828) 100 weight section and a photopolymerization nature monomer. The dicyandiamide 20 weight section was carried out as the epoxy acrylate (Showa High Polymer Co., Ltd. make, RIPOKISHI VR-60) 15 weight section and a curing agent, stirring mixing of the detailed silica (product [ made from Japanese Aerosil ], Aerosil R972) 5 weight section, the spherical silica (ADOMA tex company make, SO-C4) 15 weight section, and the photopolymerization initiator (Ciba-Geigy make, IRUGA cure 651) 2 weight section was carried out as an inorganic bulking agent, it kneaded with 3 more roll, and the adhesives constituent was obtained.

[0018] Next, the spherical silica spacer with a diameter of 6 micrometers was mixed 1% to this adhesives constituent, and the liquid crystal cell was produced in the following ways.

(Sealant spreading) The pattern of a square with a line breadth of 0.3mm is screen-stenciled on the glass substrate with ITO in which the orientation film was made to form using the stainless steel screen version of 250 meshes (one-side square of 3cm).

(Optical exposure) High-pressure-mercury-lamp 10-second exposure (2000 mJ / cm<sup>2</sup>)

(Lamination, heat hardening) Where lamination and the pressure of 1kg/cm<sup>2</sup> are put for the direction of orientation to become 90 degrees to the orientation processing direction of the substrate which printed the sealant about the glass substrate with ITO in which the orientation film was made to form, heat hardening was carried out for 150 degrees C in hot air drying equipment / 120 minutes.

(Liquid crystal impregnation, obturation) Cyano \*\*\*\*\* (the Merck Co. make, ZLI-1132) was poured in, and the inlet was obturated with acrylic UV hardening resin.

In the above-mentioned point, the seal pattern was crushed by the optical exposure after sealant screen-stencil by sticking another substrate by pressure, although the crosslinking reaction of

epoxy acrylate starts and a sealant front face is in a tuck free-lancer's condition, and two substrates were fixed.

[0019] Evaluation performed the item shown below.

(1) Adhesive strength after heat hardening (a glass substrate with a knife tearing off). (2) The result of pressure cooker test (after processing liquid crystal cell for 24 hours under 125-degree-C / 100%RH / 2.3 atmospheric pressures, square wave of \*\*3V is impressed and nonuniformity of display is evaluated) evaluation is as being shown in Table 2.

[0020] (Example 2-6) The sealant constituent was prepared by the presentation shown in Table 1 by the same actuation as an example 1, and the liquid crystal cell was produced in the same procedure as an example 1. For the result of Table 1 and evaluation, the contents of the constituent and cel production conditions are as being shown in Table 2. (Constituent is the same as that of an example 1 among cel production conditions like an example 1 except a heat hardening process except inside, the hardenability resin made from heat, a radiation and/or a photopolymerization nature monomer, and a curing agent)

[0021]

[Table 1]

表1

| 実施例 | シール材組成物（重量部）                                                                  |                                                            |                                      | セル作製条件     |
|-----|-------------------------------------------------------------------------------|------------------------------------------------------------|--------------------------------------|------------|
|     | 熱硬化性樹脂                                                                        | 放射線及び／又は光重合性モノマー                                           | 硬化剤                                  |            |
| 2   | ビスフェノールA型エポキシ<br>エピコート828（油化シェル） 100                                          | ウレタンアクリレート<br>AT-600（共栄社化学） 20                             | アジピン酸ジヒドラジド 15                       | 150°C／120分 |
| 3   | ビスフェノールA型エポキシ<br>エピコート828（油化シェル） 80<br>ナフタレン型エポキシ<br>HP-4032（大日本インキ） 20       | エポキシアクリレート<br>リボキシSP-1509H（昭和高分子） 30                       | アダクト型硬化剤<br>アミキュアNY-II（味の素） 20       | 120°C／90分  |
| 4   | ビスフェノールF型エポキシ<br>エピコート807（油化シェル） 100                                          | エポキシアクリレート<br>リボキシSP-1509H（昭和高分子） 30                       | ジシアンジアミド 20                          | 160°C／90分  |
| 5   | ビスフェノールA型エポキシ<br>エピコート828（油化シェル） 70<br>ポリエチレングリコール型エポキシ<br>エポライト40E（共栄社化学） 30 | エポキシアクリレート<br>リボキシSP-1509H（昭和高分子） 30                       | マイクロカプセル型硬化剤<br>ノバキュアHX-3742（旭化成） 30 | 120°C／60分  |
| 6   | ビスフェノールA型エポキシ<br>エピコート828（油化シェル） 70<br>ポリエチレングリコール型エポキシ<br>エポライト40E（共栄社化学） 30 | エポキシアクリレート<br>リボキシSP-1509H（昭和高分子） 30<br>N,N-ジメチルアクリルアミド 10 | アジピン酸ジヒドラジド 15                       | 160°C／90分  |

[0022] (Example 7) It is as thermosetting resin as the bisphenol A mold epoxy resin (oil-ized shell epoxy company make, Epicoat 828) 100 weight section and a radiation polymerization nature monomer. Styrene The dicyandiamide 20 weight section was carried out as 15 weight sections and a curing agent, stirring mixing of the detailed silica (product [ made from Japanese Aerosil ], Aerosil R972) 5 weight section and the spherical silica (ADOMA tex company make, SO-C4) 15 weight section was carried out as an inorganic bulking agent, it kneaded with 3 more roll, and the adhesives constituent was obtained.

[0023] Next, the spherical silica spacer with a diameter of 6 micrometers was mixed 1% to this adhesives constituent, and the liquid crystal cell was produced in the same way as an example 1. However, it is a gamma ray from 60Co(es) instead of actuation of an optical exposure in the example 1. It irradiated by the dose rate of 1 Gy/s. In the above-mentioned point, although the polymerization reaction of styrene started and the sealant front face changed into a tuck free-lancer's condition by the radiation irradiation after sealant screen-stencil, by sticking another substrate by pressure, the seal pattern was crushed and two substrates were fixed. It evaluated like the example 1 and the result was shown in Table 2.

[0024] (Example 1 of a comparison) As the bisphenol A mold epoxy resin (oil-ized shell epoxy company make, Epicoat 828) 80 weight section, the o-cresol NIBORAKU mold epoxy resin (Sumitomo Chemical Co., Ltd. make, ESCN195LB) 20 weight section, and a curing agent The detailed silica (product [ made from Japanese Aerosil ], Aerosil R972) 5 weight section and the spherical silica (ADOMA tex company make, SO-C4) 15 weight section were carried out as the dicyandiamide 15 weight section and an inorganic bulking agent, stirring mixing of the methyl carbitol 10 weight section was carried out as a solvent, it kneaded with 3 more roll, and the adhesives constituent was obtained. Next, after mixing the spherical silica spacer with a diameter of 6 micrometers 1% to this adhesives constituent and applying a sealant by screen-stencil like an

example 1, the optical exposure was not performed, but prebaking was performed for 90 degrees C / 30 minutes, and the rest produced the liquid crystal cell by the same actuation as an example 1. The result of evaluation is as being shown in Table 2. When it measured by GC-MS how much the vaporization of a solvent was at the prebaking process, about 50% of the solvent contained in the constituent remained.

[0025] (Example 2 of a comparison) As the bisphenol A mold epoxy resin (oil-ized shell epoxy company make, Epicoat 828) 80 weight section, the bisphenol A mold epoxy resin (oil-ized shell epoxy company make, Epicoat 1001) 20 weight section, and a curing agent The detailed silica (product [ made from Japanese Aerosil ], Aerosil R972) 5 weight section and the spherical silica (ADOMA tex company make, SO-C4) 15 weight section were carried out as the adipic acid JIHIDORA JIDDO 20 weight section and an inorganic bulking agent, stirring mixing of the methyl carbitol 10 weight section was carried out as a solvent, it kneaded with 3 more roll, and the adhesives constituent was obtained. (Specific surface area does not contain the silica more than 5m<sup>2</sup>/g as an inorganic bulking agent)

Next, the spherical silica spacer with a diameter of 6 micrometers was mixed 1% to this adhesives constituent, and the liquid crystal cell was produced in the same way as the example 1 of a comparison. The result of evaluation is as being shown in Table 2. When the amount of residual solvents after a prebaking process was measured like the example 1 of a comparison, it turned out that it remains about 40%.

[0026]

[Table 2]

表2

|      | 加熱硬化後の接着力 | プレッシャークッカーテスト |
|------|-----------|---------------|
| 実施例1 | ガラス板破壊    | 表示ムラなし        |
| 実施例2 | ガラス板破壊    | 表示ムラなし        |
| 実施例3 | ガラス板破壊    | 表示ムラなし        |
| 実施例4 | ガラス板破壊    | 表示ムラなし        |
| 実施例5 | ガラス板破壊    | 表示ムラなし        |
| 実施例6 | ガラス板破壊    | 表示ムラなし        |
| 実施例7 | ガラス板破壊    | 表示ムラなし        |
| 比較例1 | ガラス板破壊    | 表示ムラあり        |
| 比較例2 | ガラス板破壊    | 表示ムラあり        |

[0027]

[Effect of the Invention] The liquid crystal display component which the productivity of a liquid crystal display component and the yield of the liquid crystal display component using the manufacture approach of the sealant for liquid crystal display components and liquid crystal display component which were shown by this invention improve compared with a conventional liquid crystal sealant and the conventional manufacture approach, and is excellent in dependability is offered.